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Development of a Scalable Process Control System for Chemical Soil Washing to Remove Uranyl Oxide

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Abstract

The U.S. Army has responsibility for maintaining or managing a large number of facilities that are or have been used for training troops and developing/testing equipment and munitions, including ranges that may have been contaminated with uranium. Licenses issued by the Nuclear Regulatory Commission (NRC) for use of radiological materials such as depleted uranium (DU) specify the isotopes that can be used, along with possession limits for the site. U.S. Army Engineer Research and Development Center (ERDC) researchers have developed a soil washing system to leach DU oxides from soil. The Institute for Clean Energy Technology (ICET) at Mississippi State University (MSU) has developed an effective survey system to accurately locate areas of DU contamination for removal and disposal. The ICET also has a history of developing control systems for sophisticated test beds. ICET has combined its experience in development of control systems with DU detection methods to develop a process control system for the ERDC soil leaching system for extracting DU from contaminated range soil. The ICET system control and data acquisition (SCADA) system has been demonstrated to control pumps and valves, maintain leaching solution chemistry to user-defined setpoints, and detect environmental levels of DU oxides in leachate. The SCADA system will assist the ERDC Environmental Laboratory (EL) in transitioning development of the soil washing system from pilot to a full-scale system.

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Preface

This report describes work conducted for the U.S. Army Engineer Research and Development Center (ERDC), Environmental Laboratory (EL), Environmental Quality/Installations (EQ/I) Research Program project entitled "Depleted Uranium (DU) Munitions & Munitions Residues Management for Range Sustainability and Legacy Sites.". The work was funded via Battelle Memorial Institute Contract W911NF-11-D-0001 TODO 0184 by Mississippi State University (MSU) under Purchase Order US001-0000370554.

This report documents the development of a prototype flexible and scalable process control system with strategically positioned radiological sensors for monitoring and automating the DU leaching process developed by the ERDC-EL. Dr. Steven L. Larson and John H. Ballard of the ERDC-EL, Vicksburg, MS; Jay P. McCown, Ronald J. Unz, and Dr. Charles A. Waggoner, Mississippi State University, MS; and Per Arienti, of U.S. Army Armament Research Development and Engineering Center (ARDEC), Picatinny Arsenal, NJ, prepared this report. In-house review was provided by Deborah Felt and Roy Wade of the Environmental Engineering Branch (EP-E).

This focus area is under the direct supervision of John Ballard, Assistant to the Technical Director, ERDC-EL, and under the general supervision of Dr. Elizabeth Ferguson, Technical Director for Military Materials in the Environment, ERDC-EL. Dr. Jack Davis was Deputy Director, ERDC-EL, and Dr. Beth Fleming was Director, ERDC-EL. LTC John T. Tucker III was the Acting Commander of ERDC, and Dr. Jeffery P. Holland was Director of ERDC.

Executive Summary

The U.S. Army has responsibility for maintaining or managing a large number of facilities that are or have been used for training troops and developing/testing equipment and munitions. These sites include ranges that may have been contaminated with heavy metals such as lead, tungsten, or uranium. Developing, testing, and training with weapon systems employing these materials should not and cannot be avoided. Therefore, it is imperative that cost-effective and environmentally effective techniques be developed and made available to maintain ranges where these weapon systems are used.

Licenses issued by the Nuclear Regulatory Commission (NRC) for use of radiological materials such as depleted uranium (DU) specify the isotopes that can be used along with possession limits for the site. Activities associated with maintaining an NRC license for individual ranges are expensive, regardless of the activity level on the range. Meeting the strict NRC guidelines for clearing ranges of DU contamination would allow licenses to be canceled, a cost-saving measure. The infrastructure and technical capability of ERDC and ICET can be beneficially and cost-effectively used to reduce the expense of clearing ranges where DU has been used.

ERDC researchers have developed a soil washing system to leach DU oxides from soil. The Institute for Clean Energy Technology (ICET) at Mississippi State University (MSU) has developed an effective survey system to accurately locate areas of DU contamination for removal and disposal. ICET also has a history of developing control systems for sophisticated test beds. ICET has combined its experience in development of control systems with DU detection methods to develop a process control system for the ERDC soil leaching system for extracting DU from contaminated range soil.

A scalable process control system for the ERDC chemical soil washing to remove uranyl oxides has been developed by ICET. An Excel-based model has been used to validate logic sequences and aid in design of physical infrastructure to validate performance capability of the control system. The ICET system control and data acquisition (SCADA) has been

demonstrated to control pumps and valves, maintain leaching solution chemistry to user-defined setpoints, and detect environmental levels of DU oxides in leachate.

The SCADA system will assist ERDC in transitioning development of the soil washing system from laboratory scale to proof of concept and full scale. It has been developed using industrial architecture, software, and programmable logic controllers. The system can be modified to control an array of small leaching heaps as well as a large-scale soil washing system.

Unit Conversion Factors

Multiply	By	To Obtain
cubic feet	0.02831685	cubic meters
cubic yards	0.7645549	cubic meters
degrees Fahrenheit	(F-32)/1.8	degrees Celsius
gallons (U.S. liquid)	3.785412 E-03	cubic meters
inches	0.0254	meters
pints (U.S. liquid)	0.473176	liters

Acronyms

APG	Aberdeen Proving Ground
ARDEC	Armament Research, Development and Engineering Center
BRAC	Base Realignment and Closure
cph	counts per hour
cpm	counts per minute
CWA	Clean Water Act
DHS	Department of Homeland Security
DoD	Department of Defense
DOE	Department of Energy
DU	Depleted Uranium
EL	Environmental Laboratory
EPA	Environmental Protection Agency
ERDC	Engineer Research and Development Center
FEMA	Federal Emergency Management Agency
GUI	Graphical User Interface
ICET	Institute for Clean Energy
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MSU	Mississippi State University
NORM	Naturally Occurring Radioactive Materials
NRC	Nuclear Regulatory Commission
NU	Natural Uranium
ORP	Oxidation-Reduction Potential
RCRA	Resource Conservation and Recovery Act

RDD	Radiological Dispersive Device
RSSLS	Research Scale Soil Leaching System
SCADA	System Control and Data Acquisition
USACE	United States Army Corps of Engineers
YPG	Yuma Proving Ground

1 Introduction

The U.S. Army has responsibility for maintaining or managing a large number of facilities that are or have been used for training troops and developing/testing equipment and munitions. These sites include ranges that may have been contaminated with heavy metals such as lead, tungsten, or uranium. Many types of weapon systems such as mines, mortars, grenades, and rockets contain energetic material that can represent a severe danger to personnel and complicate recovery or disposal of heavy metal contamination. Developing, testing, and training with weapon systems employing these materials should not and cannot be avoided. Therefore, it is imperative that cost-effective and environmentally effective techniques be developed and made available to maintain ranges where these weapon systems are used.

Management of ranges where weapon systems are used can be subject to regulation and/or licensure from both the U.S. Environmental Protection Agency (US EPA) and the Nuclear Regulatory Commission (NRC). Small weapons firing can result in contamination of ranges with lead, raising concern about risks to human and environmental health. Material contaminated with lead above threshold levels is subject to management under both the Clean Water (CWA) and Resource Conservation and Recovery Acts (RCRA). Likewise, material contaminated with explosive substances is also subject to management under the RCRA and possibly the CWA.

Variations of tungsten and depleted uranium (DU) alloys have been evaluated and used as armor penetrating projectiles. Both of these metals require management of ranges where they are, or have been, used. All isotopes of uranium are radioactive, including DU, which is almost exclusively U-238. The Army currently is dealing with licensure issues associated with ranges where DU has been historically used. Additionally, ranges at both the Aberdeen Proving Ground (APG) and Yuma Proving Ground (YPG) have been licensed for development and testing of DU munitions.

Licenses issued by the NRC for use of radiological materials such as DU will specify the isotopes that can be used along with possession limits for

the site. Use restrictions can be imposed, and licenses will include inspection and management responsibilities. Activities associated with maintaining an NRC license for individual ranges are expensive, regardless of the activity level on the range. Proposed changes to the classification of these soils for licensure are currently being considered by respective regulatory groups. The Army Range Technology Program (ARTP) initiative was established to

1. design and evaluate sensor systems for the detection of munitions-related DU material and discrimination of such from naturally occurring radioactive materials (NORM)
2. design and evaluate technologies for the physical separation, when required, of DU from range soils or water at Department of Defense (DoD) facilities.

A key to sustaining DoD's operational ranges is the ability to locate, contain, or when required, remove DU without generating large quantities of waste.

U.S. Armed Forces benefit from having access to the most effective weapon systems that developers and military personnel can assemble. Testing the munitions is an integral part of the development process. Range managers benefit by having access to the tools developed under the ARTP. In addition, the ARTP, by developing and applying scientifically defensible low-cost range maintenance practices, will support DoD efforts in implementing measures protective of human health and the environment.

DU alloys oxidize in the environment and may migrate from the original location, causing the spread of DU contamination. Current DoD policy prohibits the firing of DU for training or testing. Firing is now only allowed into range catch boxes. The use of catch boxes aids in containment and ease of recovery of the DU material. Historically, DU was fired into both catch boxes and on open firing ranges; therefore, contamination at DoD sites varies.

It is critical to maintain the capability to test DU rounds undergoing improvement or development. Sustainable range management requires knowledge of the presence and extent of DU on a range, and range management practices often call for locating, containing, and, when appropriate, recovering fired penetrators. The recovery process can be

complicated by the fact that metallic uranium is reactive, oxidizing once it has been deposited into soil. The recovery process needs a variety of measurement systems for precisely locating the DU material, facilitating recovery of DU with a minimal quantity of contaminated soil, and controlling process equipment used to separate DU from soil and associated debris.

Bases with ranges where ARTP-type technologies are used may also have concerns associated with contamination with volatile organic pollutants. This contamination can include greasing solvents such as trichloroethane petroleum products from leaking underground storage tanks or numerous other pollutants. Many of these bases have been removed from active inventory by base closure and realignment actions. Transition of these bases from Army ownership to private development often hinges on resolution of such environmental issues.

The U.S. Army Corp of Engineers (USACE) and the U.S. Army Engineer Research and Development Center (ERDC) are called on to support Army activities at bases and ranges, particularly in matters associated with environmental management. It can also be pointed out that the USACE is called upon by the Federal Emergency Management Agency (FEMA) to manage the disposal of massive amounts of debris from disasters such as Hurricane Katrina. These activities can involve discrimination between waste forms with respect to proper disposal methods. Debris that contains radiological materials (smoke detectors, soil moisture gauges, medical imaging material, etc.), toxic or hazardous materials such as pesticides or asbestos, consumer products such as refrigerators or automobiles, and even demolition material such as gypsum board are subject to disposal in appropriate manners.

The United States has lived under the threat of terrorist acts for nearly 2 decades. One of these threats has been the detonation of a radiological dispersion device (RDD), otherwise known as a *dirty bomb*. A technology that can discriminate between debris contaminated above threshold levels with radiological material from other debris would aid government agencies involved in disaster mitigation efforts.

The contribution of Mississippi State University (MSU) to the ARTP effort has been focused on evaluating detection and measurement technologies that can rapidly and accurately detect and locate DU residue (e.g.,

fragments, penetrators) from the use of munitions that contain DU (Wang et al. 2008). Notably, much of the previous field work identifying locations of radiological contamination has been conducted for the purpose of designing remedial actions (Larson et al. 2009, 2012). However, there is a significant difference between conducting environmental sampling for the purpose of remediation of a site and surveying a site for the purpose of recovering fired DU projectiles. The latter case requires a much more precise determination of the vertical and horizontal location of the DU material so that the smallest amount of soil possible will be removed during excavation (Monts et al. 2009). The potential for DU corrosion products to migrate away from metallic DU penetrators must also be taken into account.

The partnership between the ERDC, MSU, and the U.S. Army Armament Research, Development and Engineering Center (ARDEC)/ARTP has proved robust for developing and delivering functional solutions to difficult problems. MSU has demonstrated its ability to provide assistance in identifying locations of elevated radiological count rates and discriminate the presence of DU on DoD ranges. The Institute for Clean Energy Technology (ICET) at MSU has a rich history of developing novel measurement systems for the DoD, the Department of Energy (DOE), the Department of Homeland Security (DHS), and private industry. The infrastructure and technical capability of MSU and ICET can be beneficially and cost-effectively used by the ERDC to address selected issues without having to develop redundant resources.

A recent life cycle cost analysis report (Walters et al. 2014) reviewed options for clearing licenses for ranges where DU has been fired and recommends a process of accurately delineating areas of contamination and removing contaminated soil to background. This process minimizes the soil and debris subject to disposal. However, additional steps to decontaminate the exhumed soil should be evaluated to further reduce costs associated with disposal and long-term liabilities.

The Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM) is a guide that describes systematic and flexible survey methods that can be used at facilities potentially contaminated with radioactive materials. The MARSSIM provides comprehensive guidance for performing surveys of contaminated land or the inside of contaminated buildings. Surveys conducted with MARSSIM provide decision-quality

data to Federal and/or State agencies for the remediation of contaminated facilities. Final-status surveys can be used to identify if facilities have been remediated or brought to release criterion.

The Base Realignment and Closure (BRAC) commission is responsible for the evaluation of post-cold war era military facilities for their sustainability and usefulness to the U.S. military. The BRAC commission provides a list of recommended facility closures to the President of the United States every 8 years. Closed military facilities must be cleared of hazardous materials prior to being released to the public. Some facilities, such as the Jefferson Proving Ground in 1988, have historically been used for evaluating the performance of DU munitions. Facilities where DU munitions have been fired must have enough of the contaminated soil remediated such that radiation levels approach those of the naturally occurring radioactive materials (NORM). Regions of contamination can be found by using data collected during systematic MARSSIM-type surveys.

The ICET at MSU has previously developed technology to perform MARSSIM-type surveys of contaminated facilities. ICET has a variety of survey systems and radiation detection instrumentation to provide facilities with decision-quality data. The surveying infrastructure at ICET includes the following:

- 3-meter-wide motorized surveying system
- 1-meter-wide motorized surveying system
- several half-meter, man-powered surveying systems
- portable hyper-pure germanium detector
- large variety of handheld surveying units and gamma ray scintillation detectors.

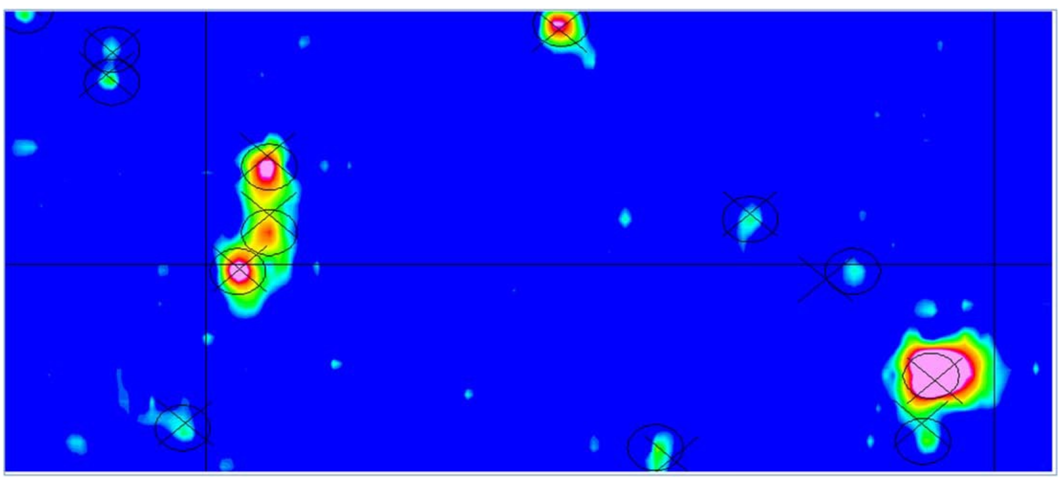
ICET also has a fully equipped counting laboratory for the evaluation of radioactive samples. Photographs of the 1-meter and 3-meter motorized surveying system can be seen in Figure 1.

The motorized and man-powered surveying systems are equipped with gamma ray scintillation detectors for radiation monitoring and high-precision GPS receivers for recording position. The surveying systems are used for performing very accurate surveys of large areas. These systems have been evaluated for a full range of operator safety categories by the Health and Safety Office at YPG. Safety officers placed no limits to on-site

surveying using these motorized and man-powered surveying systems. Postsurvey data processing can provide accurate maps of the surveyed areas and a list of contamination areas. Postprocessing analysis can locate the center of contamination within 4 inches. Figure 2 displays an example of survey data and locations of contaminants from a previously conducted survey with the 1-meter-wide surveying system.

Figure 1. Photographs of the 1-meter-wide surveying system (left) and the 3-meter-wide surveying system (right).

Figure 2. Example of survey data.



The ability to accurately locate DU contamination can be used in simplifying the clearing process of facilities. Historically, the top soil is completely removed and disposed of during the remediation process. The high-precision survey data can be used to identify areas where soil needs to be removed while leaving behind uncontaminated soil. Removing only contaminated soil greatly reduces the volume of soil required for disposal. This reduction in soil volume also reduces the cost of disposal by orders of magnitude (Walters et al. 2014). Figure 3 shows examples of the process of soil excavation during postsurvey remediation.

Figure 3. Postsurvey soil removal.

Researchers at ERDC (Vicksburg, MS) have developed a method for leaching uranium from contaminated soil fines. Following removal of DU fragments by physical separation (Larson et al. 2012), the soil fines remain contaminated by tightly bound DU oxides. The leaching system removes uranium from these soil fines and deposits the uranium into a fishbone aggregate. The previously contaminated soil can be redistributed at the original contamination site once remediated. The contaminated fishbone aggregate then needs proper disposal. It is important to note that the volume of aggregate for disposal will be significantly less than that of the soil remediated. This reduction in volume of contaminated material reduces the cost of remediation by approximately one additional order of magnitude (Walters et al. 2014).

2 System Design Considerations

2.1 General considerations

The soil washing system developed at the ERDC Environmental Laboratory (EL) employs acetic acid-acetate dissolution of uranyl oxides (Larson et al. 20012). These oxides, along with others extracted from the soil matrix, are then removed from solution using one or more adsorption or ion exchange materials. Efficiency of DU leaching is dependent on maintaining favorable pH, oxidation-reduction potential (ORP), and acetate ligand concentration. Soil factors can adversely influence leaching chemistry, so the control system for the automated process must be capable of continuously monitoring leaching solution conditions and adding reagents to maintain the desired chemistry.

ICET has extensive experience in developing test beds and control systems to evaluate chemical and/or physical processes. This expertise has been applied to development of a small-scale, yet highly scalable, system control and data acquisition (SCADA) system. The SCADA system will facilitate scale up and performance evaluation/improvement of the ERDC-EL soil washing process.

The SCADA demonstration scale unit developed by ICET is intended to serve a variety of purposes. The system has the capacity to operate soil-leaching systems at a variety of points during the scale-up processes of research and development (R&D). Future scale-ups will likely require more sensors and sensor locations as the effectiveness of the soil washing process is optimized. Data logging will need to be done with more granularity during R&D compared to that of a functioning production unit. Different types of sensors may be utilized during the R&D process in order to identify the most effective minimal suite of sensors necessary to control the production units. Initial research activities will require having an array of small leaching units to run in parallel in order to parametrically evaluate heap geometry, soil-type influences, effect of filter aids to increase leaching flow rates, and factors affecting leaching kinetics. Finally, logic sequences may need to be easily changed for individual leaching vessels from the user interface during these parametric studies.

A variety of heap geometries and soil types needs to be studied in order to accomplish sizing of pumps, leaching delivery rates, etc. This also implies the need for flexibility with regard to the ability to control pumps of different sizes, types, and manufacturing sources.

Leach generation rate will be a function of leaching solution head pressure, soil type, and heap depth. Additional factors influencing leaching rates and uniformity include the presence of rocks or debris within the soil column. Studies will need to include soil screening to remove impediments to uniform leaching of the heap along with the use of soil amendments to increase leaching rates (Larson et al. 2009, 2012).

The following set of design parameters has been discussed with the ERDC-EL research team to establish performance parameters for the control system. Fluid flow through the system has a major impact on a variety of parameters. Depth of the leaching heap will be dependent on the hydraulic conductivity of the soil. This impacts either the mass of soil that can be treated at one time or the footprint of the leach heap. Hydraulic conductivity and the footprint of the heap will effectively dictate delivery rate of the leaching solution and the rate of leachate production. These rates directly impact sizing of pumps, volume requirements for the leaching solution, along with the size of leaching solution and leachate collection vessels.

A primary design consideration for the overall system is identification of parameters necessary for successfully washing the range of heap sizes that may be required at various ranges. The heap size offering optimal economy of scale will vary as a function of the overall volume of soil to be treated and leaching characteristics of the soil at that site. Variability of soil characteristics at a single site can result in large differences in the size of a heap. For instance, as reported in Larson et al. (2009), the catch box on Range 17A of YPG is construction sand—highly porous with a high hydraulic conductivity. Range soil at YPG is a mixture of pebbles, rocks, sand, and very fine soil particles. It is likely that range soil will require a filtering aid to increase hydraulic conductivity. This will have a significant impact on the soil batch size to be treated. Production units of this technology will need to be scaled for each site and/or involve use of procedures specific to soil types and characteristics. Scalability of the SCADA must accommodate infrastructure required for heaps of a few cubic yards to 100 cubic yards.

2.2 System components

2.2.1 Heap(s)

The kinetics of leaching DU oxides from soil has not been established beyond bench scale. This will include evaluating the optimum soil depth of heaps as a function of physical and chemical characteristics of the soil. Also to be considered will be the oxidation state/mineral form of the DU oxides to be removed. Initial research activities will involve parallel leaching studies employing heaps of 1 to 2 cubic feet (ft^3). However, it is also possible that a suite of leaching heaps may be required to deal with soils of low hydraulic conductivity.

The SCADA design needs to include the potential for a single control unit to oversee operation of numerous leaching processes. This may involve dedicated leaching solution and leachate collection/processing infrastructure or use of centralized leaching solution and leachate processing infrastructure.

2.2.2 Spray fields

Each heap will require its own leaching solution spray or network of piping. It is likely that an optimum height of leaching solution covering the heap will be used to provide a hydraulic pressure head to increase the rate of leachate production. The SCADA design needs to accommodate controlling optimum delivery of leaching solution to the heap, regardless of the method selected for delivering the solution to the heap.

2.2.3 Pumps

A variety of pump types will be needed to equip both research-scale and full-scale leaching systems. The leaching system scale will influence the size of pumps and piping, but it can also influence how they are controlled. On/off switches will control most pumps. However, it is likely that larger systems will also include pumps controlled by variable frequency drives. The SCADA must be capable of controlling the full spectrum of pump types that will be employed throughout the complete range of process scales.

The number of pumps used in different scales of the leaching system will also depend on management of both the leaching solution and leachate. The SCADA will be developed to accommodate either a batch process for

conducting leaching and processing of leachate or a continuous cycling of leachate to the leaching solution tank. Both processes will be coupled with continuous adjustment of the leaching solution chemistry to maintain setpoints for solution pH, conductivity, etc.

2.2.3.1 Mixing

Tank mixing will be more appropriately accomplished using pumps than mechanical mixers. Tank mixing pumps for full-scale systems will probably be controlled by variable frequency drives to match mixing rate to the volume of solution in the tank. Piping used for continually circulating liquid in leaching solution tanks, leachate collection tanks, and waste tanks also serves as an ideal location for chemistry sensors. Piping loops for tank mixing can also serve as a manifold for valving leaching solution to various heaps and eliminating the need for multiple pumps to service a suite of small heaps.

2.2.3.2 Spray field(s)

Pumps may or may not be needed to provide leaching solution to the leaching heaps. Properly sized pumps for tank mixing can produce sufficient volumetric flow in the mixing loop to supply leaching solution to the heap via a control valve.

2.2.3.3 Chemistry concentration

Leaching-solution chemistry will be controlled by addition of concentrated reagents and or water. Addition of reagents to the leaching solution is to be monitored to allow precise measuring of individual chemical constituents. Peristaltic or gear pumps are candidates for calibrated delivery of concentrated reagents to the leaching solution.

2.2.3.4 Leachate transfer

Leachate collected from the heap will require two filtering processes, one physical and the other chemical. Leachate will contain some level of suspended solids that must be removed before removing dissolved DU oxides by the fishbone apatite. Sizing of the leachate transfer pump will depend on the

- volumetric flow needed to facilitate efficient capture of the dissolved DU oxides by the apatite filter

- combined maximum operating differential across the combined filtration units
- piping and valve network transferring fluid to either the waste tank or the leaching solution tank.

2.2.3.5 Heap/Piping rinse

The greatest economic impact of the ERDC soil washing system will be achieved through sufficient removal of DU radiological activity to allow placement of the processed soil back to its original site, based on current and proposed regulatory levels. However, re-placement will require washing the processed heap material to remove, or denature, the process chemistry. It is also possible that offsite disposal of the processed soil will still require removal/denaturing the leaching chemistry. Either option, then, requires transfer of liquids from the heap to a waste tank.

Use of a two-solution leaching process will also require rinsing the heap and clearing lines of the previous leaching solution. This set of activities will also generate waste liquids. Pumps and valving will be needed to use rinse water/fluids to accomplish flushing the piping and washing the heap.

2.2.3.6 Waste transfer

Waste fluids generated from rinsing lines or the heap will be stored in a waste tank. A pump will be required to transfer the waste fluids to containers, or a mobile tank, for offsite disposal.

2.2.4 Valves

Extensive use of valves will be required to control the flow of fluids. The scale of the system and specifics of the leaching process will dictate the number of pumps and valves needed to achieve process control. System development will incorporate the most efficient, dependable, and cost-effective combination of pumps and valves. Initial design considerations for the SCADA include recognition of the flexibility of expansion that will be required to accommodate control of the range of options that may occur.

2.2.5 Sensors

A variety of chemical, physical, and radiological sensors are needed to control the leaching process. The ERDC-EL DU leaching process employed

a single leaching solution as this control system was under development. However, the potential existed to alternate between two leaching solutions. Therefore, the control system, SCADA, has been developed to incorporate two leaching solutions and a water rinse/wash before switching solutions.

Sensors listed below will provide control measurements for the single leaching solution design. Additional chemical sensors may be required to monitor composition of the second leaching solution. Design of the SCADA must incorporate consideration for expanding the number and types of chemical sensors beyond the initial suite of pH, ORP, and conductivity.

2.2.5.1 pH

Leaching solution pH and total acetate concentration in solution are important elements for achieving dissolution of DU oxides. Industrial pH electrodes have been employed in the chemical industry for decades. These units are designed to maintain calibration during long-term function in caustic or corrosive environments.

2.2.5.2 ORP

The ORP for the leaching solution is another important element necessary to achieve efficient dissolution of the DU oxides. Industrial electrodes are also available for this application.

2.2.5.3 Conductivity

The leaching process will extract a variety of soluble ions from the heap. Conductivity of the leaching solution and leachate will reflect changes in total ion/dissolved solids concentration in these solutions. Conductivity needs to be monitored and correlated to total acetate concentration in order to estimate acetate losses and/or other dissolved salts in solutions.

2.2.5.4 Radioactivity

All isotopes of uranium are radioactive; however, U-238 is an alpha emitter. This means that it is very difficult to directly monitor U-238 activity in a flowing system. However, protactinium, Pa-234m, is a short-lived daughter of U-238 that emits a 1001 keV gamma. This isotope is routinely used as an easily monitored surrogate for U-238 during

environmental surveys. The concentration/activity of uranium in the leachate will be monitored using a gamma detector.

There are numerous naturally occurring radioactive materials in soil. ICET has developed a spectral ratio methodology for specifically monitoring U-238 activity in soil. This technique also employs a spectral ratio technique for U-238 to Bi- 214 to discriminate between U-238 activity levels that are naturally occurring and those that result from the presence of DU. The ICET DU monitoring methodology uses lanthanum bromide detectors to achieve sufficient energy resolution of gamma radiation to discriminate between naturally occurring uranium and activities that include DU. A lanthanum bromide detector will be positioned before and one after the fishbone apatite adsorption column to monitor its collection efficiency. A reduction of removal efficiency will signal time to replace the exhausted adsorption medium.

2.2.5.5 Specific ion electrode(s)

Alternative leaching chemistry can require specific ion electrodes to monitor and maintain optimal chemistry of the leaching solutions. The prototype system developed for proof-of-concept testing will not include specific ion electrodes or other analytical tools such as UV-Visible detectors. However, the SCADA design will accommodate scale up to include these options.

2.2.5.6 Flow

Two types of flow indicators can be used in development of the leaching system. Simple units can be employed to indicate that fluid is flowing through a pipe. Other applications can require measuring flows. Examples of measured flows include additions of concentrated chemical solutions to the leaching chemistry tank to maintain process effectiveness.

2.2.5.7 Pressure and differential pressure

Line pressure within pipes can be used to augment flow indicators to provide remote operators additional information when trouble shooting problems. Monitoring the status of filters like the ones removing suspended solids from leachate requires differential pressure monitors.

2.2.5.8 Liquid height

There are numerous applications of liquid-height-level monitors within the soil washing process. Liquid levels in tanks need to be monitored for minimum, maximum, and overflow conditions. Liquid levels above heaps need to be monitored to determine when to add additional leaching solution.

2.2.6 Graphical user interface (GUI)

The operating system for this chemical process must provide user ease and flexibility to control or modify operation. The GUI will include screens that display the status of operating hardware, graphs of trend lines for monitoring chemical or physical parameters, visual and audible alarms for identifying sources of problems, and lists of setpoints and acceptable ranges for all operational parameters.

The GUI for the prototype SCADA developed by ICET assumes batch operation of the leaching process, a single leaching solution, and a single leaching heap. All screens developed for this version of the SCADA are designed to operate a proof-of-concept system of tanks, sensors, pumps, and vessels.

2.2.6.1 User input (Setpoints)

A screen or a set of screens will be available for the system operator to define setpoints for all operational parameters. This will include the ability to define the acceptable operating range as well as alarm levels.

2.2.6.2 System status (Display)

The proof-of-concept system will employ a laptop computer to augment the SCADA hardware as a display for monitoring process operation. Full-scale systems will include a touch screen display connected to system electronics for on-site interrogation of process status and for changing control parameters. However, the SCADA will retain the capability to network with local or remote computers for more advanced interrogation of system operation/performance as well as for reviewing data, trend lines, etc.

2.2.6.3 Alarms

Manual or remote operation of the soil leaching system will require alarms to notify operators that a setpoint has been violated or a problem has arisen with the system. Alarms are particularly important to remote operation of the system. Examples of alarms can include the following:

- loss of flow due to broken piping
- piping pressure levels below acceptable levels
- inability to maintain required leaching solution head on heap
- insufficient fluid levels in tanks or tank overflow conditions
- loss of DU removal efficiency of the adsorption filter due to exhaustion
- solution chemistry out of range
- failure of a pump or valve to respond.

2.2.7 Remote communications

Remote operation of systems has become commonplace and can be accomplished for remote locations via cellular communication devices. Development of the SCADA will include the potential for remote oversight of the operation.

2.2.8 Computer and electronics

The electronics components of the process control system will include a combination of a process computer and programmable logic controllers. Integration of process control logic and sensor data will be accomplished using WonderWare LonWorks software (http://global.wonderware.com).

2.2.8 Output/Reports

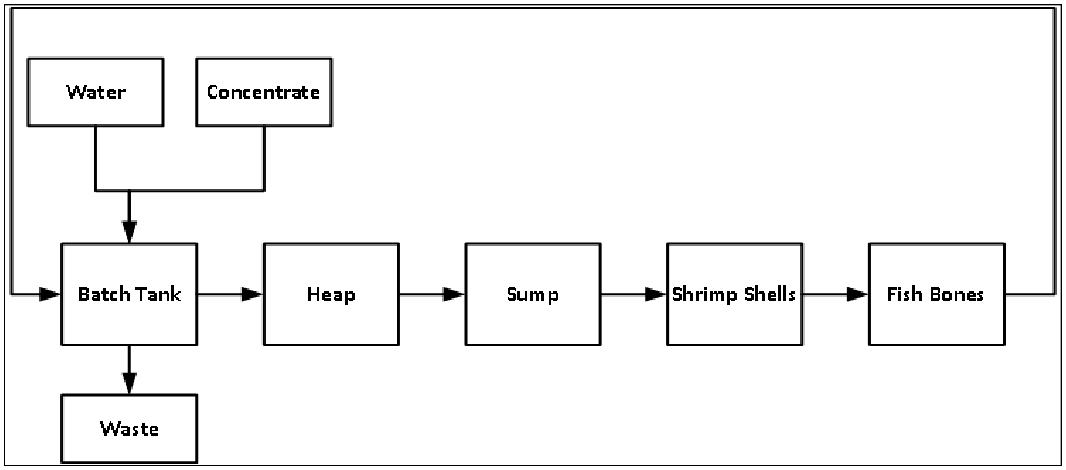
The SCADA and on-site display will provide screens for ready review of process status and trends in key parameters. Data files for all actions and measurements will be maintained and referenced to SCADA timestamps. These data files will be available for download to remote computers for incorporation into reports.

2.3 Logic sequences

Figure 4 provides a basic flow chart for the ERDC-EL DU soil leaching process. Control parameters have been identified for each unit operation and incorporated into logic sequences to accomplish process control.

Chapter 3 of this report provides a description of the SCADA and system hardware developed to demonstrate proof of concept for the control system.

Figure 4. Generalized flow diagram for the ERDC-EL depleted uranium soil washing process.



3 System Description

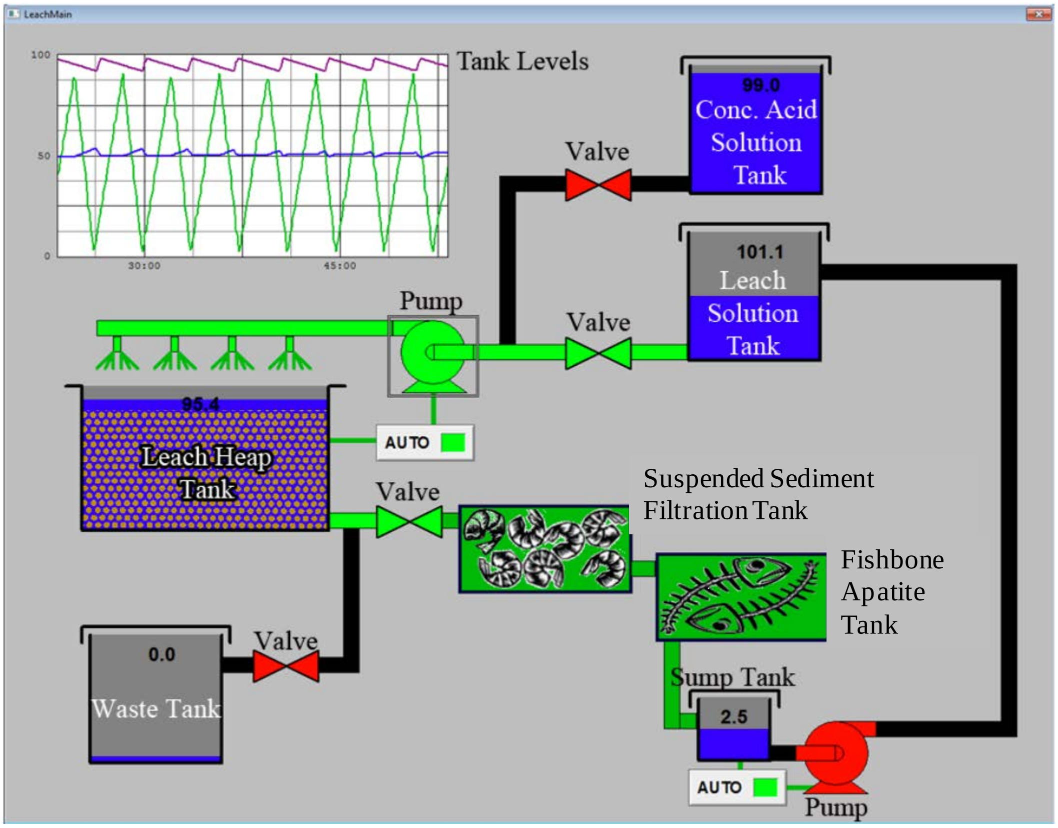
The design of the ERDC-EL DU leaching process initially included two stages for the separation of dissolved uranyl oxides from the leachate, a processed shrimp shell stage followed by adsorption by an apatite material prepared from fishbone. The current process does not include the shrimp shell phase; the ICET SCADA has retained that step because this operation served a dual purpose. Leachate passes through the Phase 1 filtration stage before going through a Phase 2 adsorption stage. The initial filtration stage would remove sediment and suspended solids in addition to removing some of the DU. It is very important to remove the fine silt and suspended particulate matter before the leachate enters the apatite adsorption column. Buildup of solids in the apatite column yields increasing differential pressure and physically blocks active adsorption sites.

Figure 5 provides a graphical representation of the proposed leaching system, including the shellfish (Phase 1 filtration) stage. The graphical representation of the system is also a GUI of a simulation used to develop and test the logic sequence used to flow fluids through the system. There are several main components to the leaching system:

- leach solution tank
- concentrated acid tank
- leach heap tank
- Phase 1 filtration tank
- Phase 2 adsorption tank, sump tank, valves, and pumps.

Logic sequences are designed to prepare the leaching solution in the leaching tank shown in Figure 5. System software adjusts the pH of the leaching solution by adding glacial acetic acid from the concentrated acid tank. Figure 5 does not show the circulation loop that allows the pump to continually mix the leaching solution tank contents. This will be more clearly demonstrated in the latter part of this chapter. The peristaltic pump used to dispense glacial acetic acid is also missing from Figure 5.

Figure 5. Simulation interface for soil leaching systems and design depiction of leaching system.

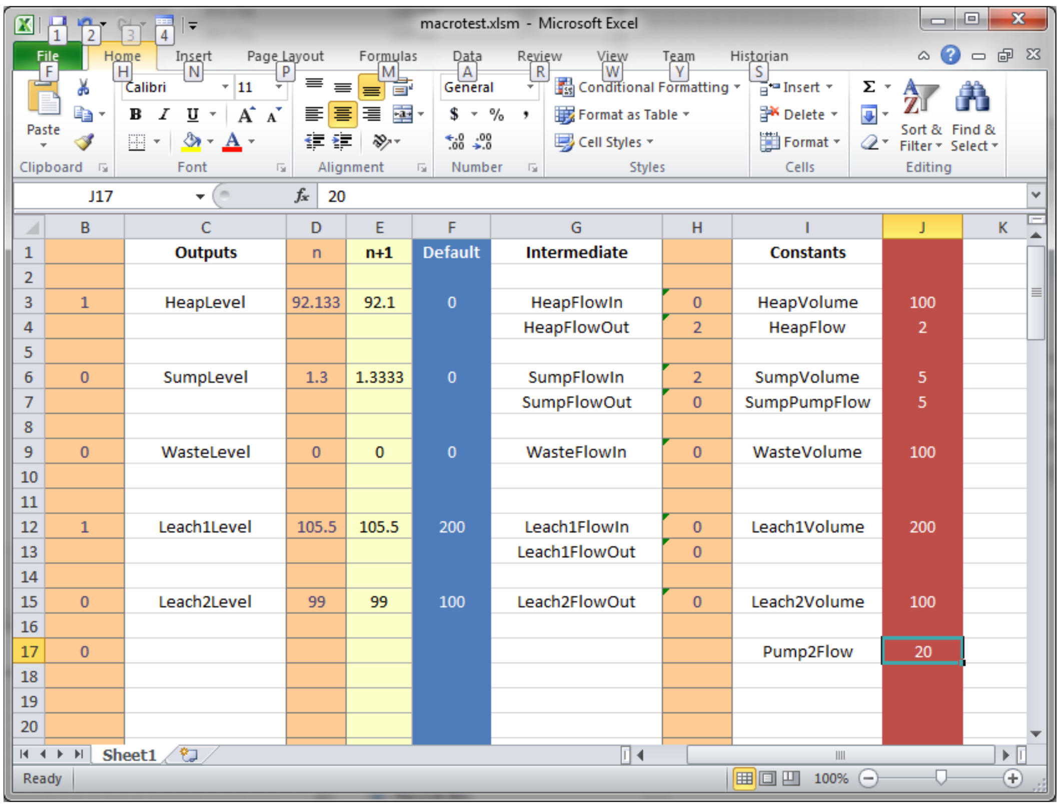


The leaching solution is delivered to the leach heap in one of two ways, either by a spray system or simply by flooding the surface of the heap. Figure 5 shows the leaching solution being sprayed onto the top of the leach heap. Either method of maintaining coverage of the heap with leaching fluid will require keeping the surface under a blanket of fluid. Height/depth of this pool will be a function of soil hydraulic conductivity.

Leachate drains through the heap and is then processed through the Phase 1 suspended sediment filtration device (indicated as shellfish aggregate), the fishbone apatite (DU adsorption), and delivered to the sump tank. The simulation monitors the volumes in the leach heap tank, sump tank, and leach solution tank and adjusts valves and pumps to maintain the system at optimum settings. The simulation is set to automatically loop the leaching fluid through the system in a batch-mode manner. Leaching solution is delivered to the heap until leaching solution tank contents reach the minimum setpoint. This activates the process of transferring contents of the sump tank back to the leaching solution tank.

An Excel spreadsheet operates in the background to conduct calculations for the flows as a function of time (Figure 6). The Excel sheet and the GUI transmit information through WonderWare using Microsoft Dynamic Data Exchange (DDE). A macroinstruction program within Excel is used to update the simulation. WonderWare frequently checks both the GUI and Excel spreadsheet for changes in pump or valve states and for calculation changes.

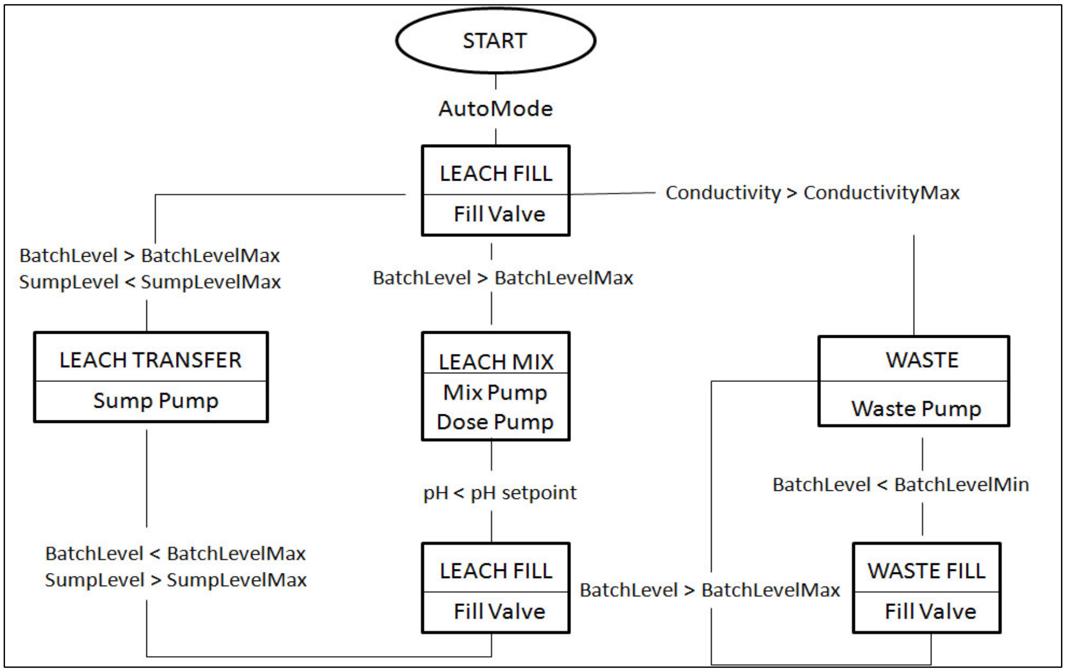
Figure 6. Screenshot of simulation Excel spreadsheet.



There are several components in the GUI that are clickable. Simulated pumps and valves that transfer fluids can be activated or deactivated by clicking on them. The simulation tracks and monitors volumes and flows but does not simulate leaching solution chemistry. Red valves or pumps indicate the valves are closed or the pumps are off. Green valves or pumps indicate the valves are open or the pumps are on. Green pipes indicate that fluids are being transferred through the indicated pipes in the simulation. Black pipes indicate no flowing of fluids.

The decision to use a batch process instead of a continuous process resulted in the implementation of the stage diagram shown in Figure 7.

Figure 7. Stage diagram of leaching process.



The process of leaching is as follows. The leaching solution tank is filled with water. Water is added until the level of the batch leaching solution reaches the user-defined fill level (BatchLevelMax). The transfer pump used to deliver leaching solution to the heap is part of a valved loop that returns fluid to the tank to continuously mix the leaching solution. Glacial acetic acid is added to the solution until the pH of the solution reaches the user-defined pH setpoint. The mixing process is identified as "Leach Mix" in the stage diagram.

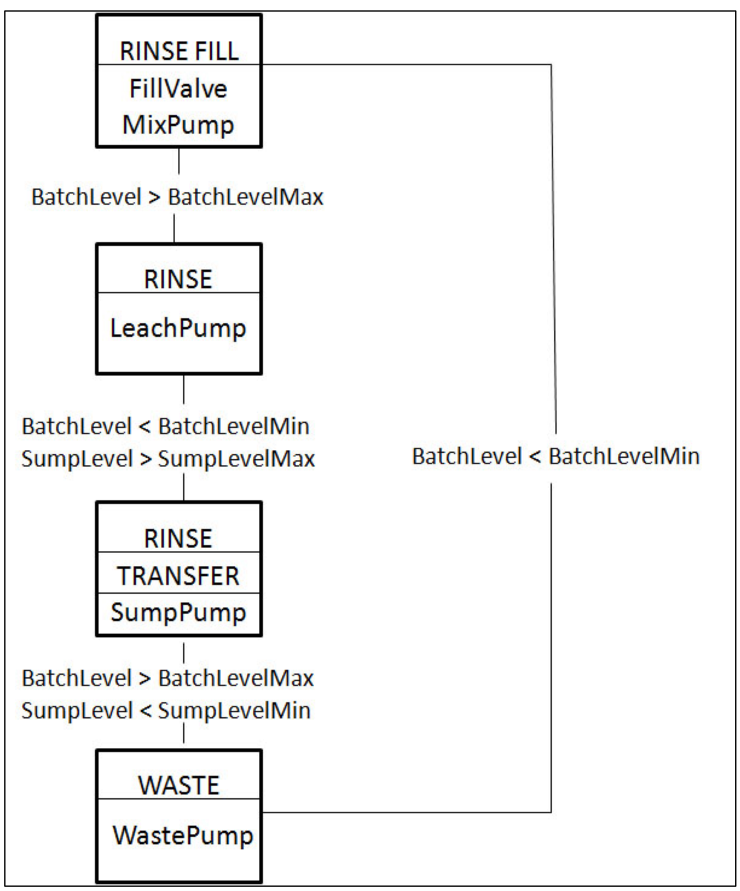
The leaching solution is then pumped to the spray bar above the leach heap ("Leach" step in the stage diagram). Software monitors the volume within the sump tank (SumpLevel) and leaching solution tank (BatchLevel). The leaching process continues until the leaching solution tank reaches the minimum level (BatchLevelMin) or until the sump tank is full (SumpLevelMax). The leachate that is collected in the sump tank is transferred back to the leaching solution tank (Leach Transfer). The process of transferring the leachate continues until the sump tank reaches the user-defined minimum (SumpLevelMin) or until the leaching solution tank is full (BatchLevelMax).

The process of Leach Fill, Leach Mix, Leach, and Leach Transfer continues until the conductivity increases to a maximum setpoint value. This indicates an increase in ionic strength outside the range of acceptability

and results in rejecting the spent solution by transferring it to the waste storage tank (Waste). The process of transferring to waste storage continues until the leaching solution tank is empty (BatchLevelMin). The leach solution tank is then filled with clean water and its chemistry is adjusted to meet user-defined criteria.

The stage diagram in Figure 8 represents steps undertaken to rinse the heap and leaching system. This will be done once the heap has been deemed clean or when clearing one leaching solution to begin using a different one. Water is added to the empty leaching solution tank until it is full (BatchLevelMax) as shown in the “Rinse Fill” stage. The pump for the leaching solution tank is turned on during the filling process to flush piping and sensors associated with the leaching solution tank can be rinsed. The rinse water is pumped to the heap in the “Rinse” stage. The process of rinsing the heap continues until leachate in the sump tank reaches user-defined chemistry/conditions. Rinse fluid in the sump tank is transferred to waste storage. The process of rinsing is repeated until the system reaches a satisfactory level of cleanliness.

Figure 8. Stage diagram of the rinsing process.



Demonstration of functionality of the process control system requires a functioning assembly of hardware to simulate the overall process. Figure 9 provides a design layout drawing for a research-scale soil leaching system (RSSLS) (Figure 10 presents a photograph of the RSSLS.). The drawing has tag IDs used for the development of the control software, and the software also contains labels for critical components. A list of tag IDs, labels, component description, manufacturer, and part number can be found in Table 1, using Figure 9 and Figure 10 for reference.

Figure 9. Schematic of the research-scale leaching system.

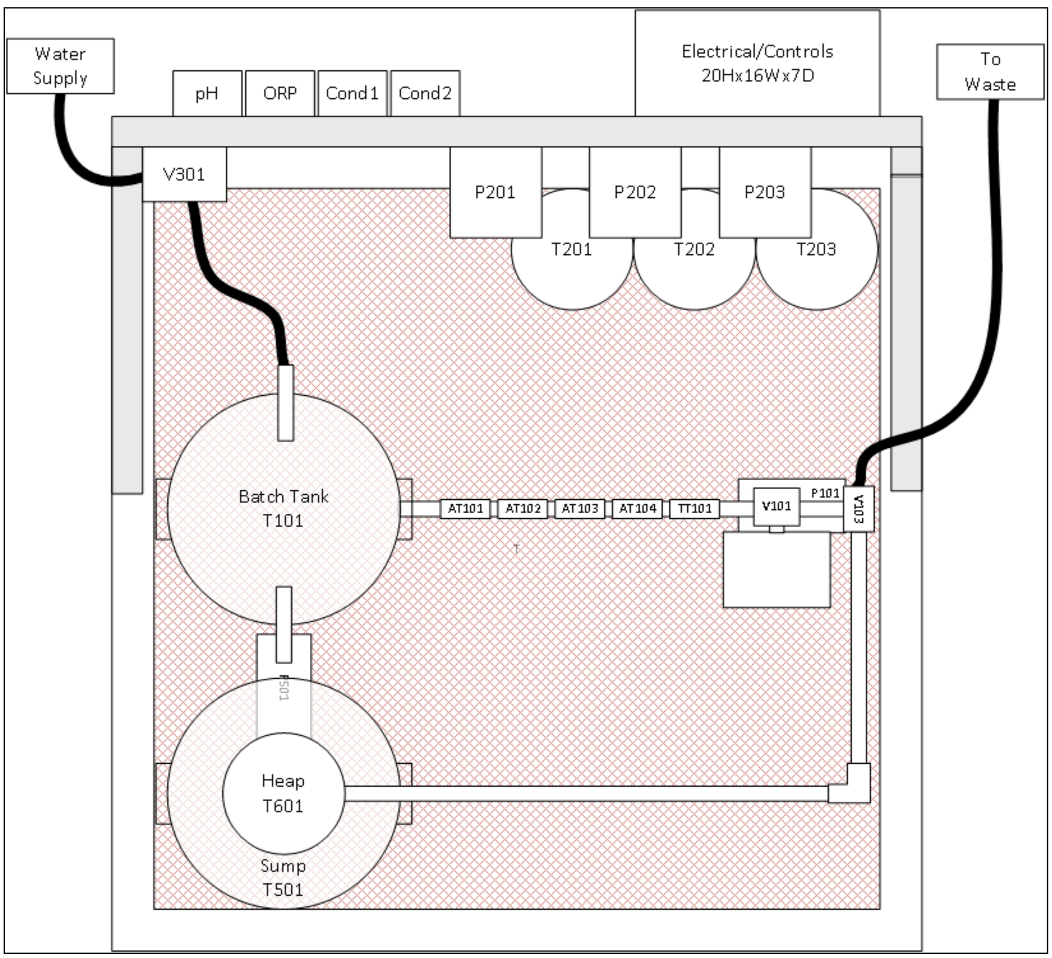


Figure 10. Photograph of the ERDC soil leaching system.

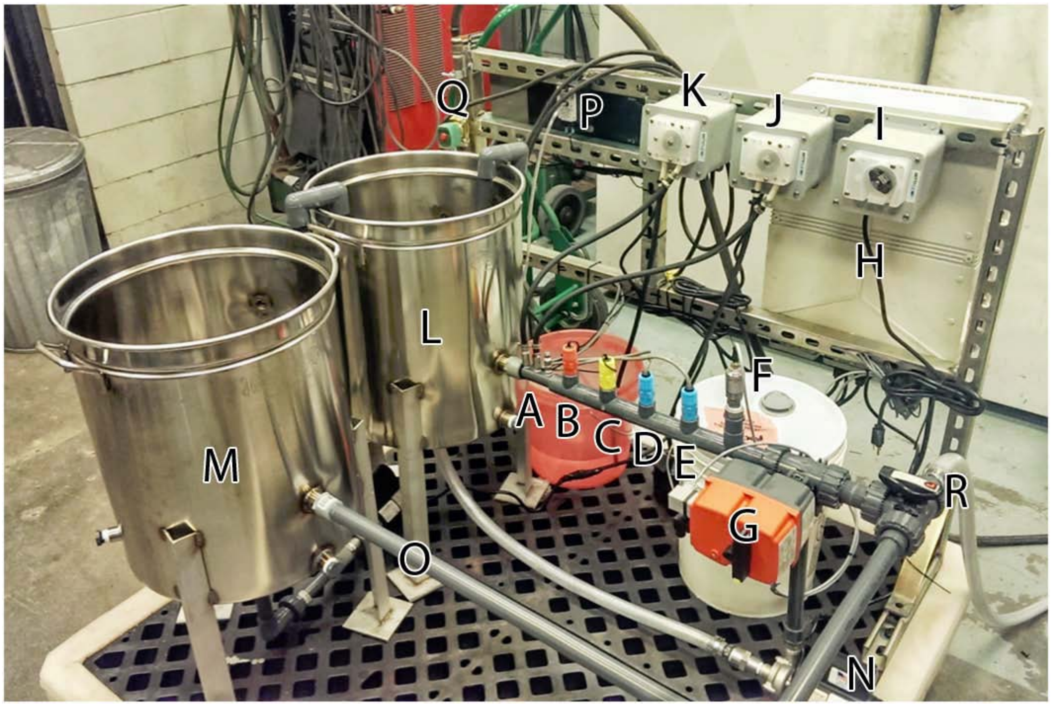


Table 1. Components of the research-scale soil leaching system.

Drawing Tag ID	Photograph Label	Description	Manufacturer	Part No.
	A	Chemical injection location		
AT101	B	pH probe	Hanna Instruments	HI 1001
AT102	C	ORP probe	Hanna Instruments	HI 2001
AT103	D	Electrical conductivity probe, 200 mS/cm	Hanna Instruments	HI 3001
AT104	E	Electrical conductivity probe, 20 mS/cm	Hanna Instruments	HI 3001
TT101	F	Temperature		
V101	G	Electrical actuated 3-way ball valve	Valworx	561406
	O,N	Stainless steel pipe		
T201, T202, T203, T601		Stainless steel tanks		
Electrical/Controls	H	Electrical and controls		
P201, P202, P203	I, J, K	Peristaltic metering pumps	Anko	OLS-11
T101, T501	L, M	62-quart stainless steel stock pot	Bayou Classic	1060
P101 P501		Stainless centrifugal pump	Cole-Parmer	WY72021-13
pH	P	Transmitters for probes	Hanna Instruments	HI 8614N

Drawing Tag ID	Photograph Label	Description	Manufacturer	Part No.
ORP	P	ORP transmitter	Hanna Instruments	HI 8615N
Cond. 1	P	Conductivity transmitter for 200 mS/cm	Hanna Instruments	HI 8936AN
Cond. 2	P	Conductivity transmitter for 20 mS/cm	Hanna Instruments	HI 8936DN
V301	Q	0.5-inch 120 vac solenoid valve	ASCO	42010
V103	R	3-way ball valve	Valworx	551406

The RSSLS is built on top of a spill containment pallet to collect any liquids splashed or leaked from components. The containment pallet also provides a solid foundation for the mounting of RSSLS infrastructure such as tank stands, pumps, and infrastructure for instrumentation and peristaltic pumps.

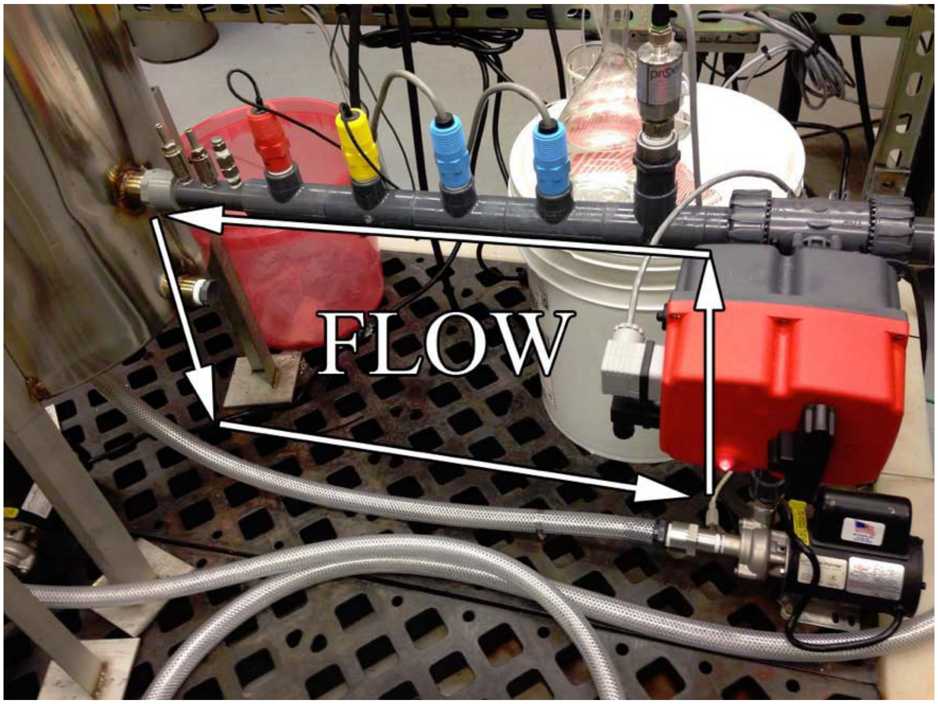
The leaching solution tank and sump tank (items L and M in Figure 10) are modified 58.7 L stainless steel pots used in the food industry. Mounting brackets, threaded bulkheads, and sensor ports have been added to each pot. Each vessel is fitted with differential pressure sensors for monitoring fluid volumes; tubing connecting the pot to the pressure sensor can be seen in Figure 11.

Figure 11. Pressure sensors for measuring tank volumes.

The leaching solution pump runs continually and is part of a piping loop that circulates solution back to the tank. Withdrawing leaching solution from the bottom of the leaching solution tank and returning it to the midway point on the side of the pot provides continual mixing of tank contents. A photograph of the plumbing and flow is shown in Figure 12. The 12 gallons per minute (gpm) centrifugal pump produces quick mixing

of leaching chemistry as concentrated reagents are added to the tank. Locating the sensor array along this loop ensures that measurements will accurately reflect the chemistry being discharged to the heap.

Figure 12. Leaching solution tank mixing plumbing.



The sensor array consists of five sensors on the return leg of the circulating loop. This is displayed in Figure 13. The five sensors measure the following chemical parameters: pH, ORP, (EC) high (200 mS/cm), EC low (20 mS/cm), and temperature. A three-way, electrically actuated ball valve is located upstream of the sensors to divert the leaching solution to the heap or to the waste tank. A three-way manual valve on the other side of the electrically actuated valve controls fluid transfer to the leach tank or to waste.

Two chemical injection locations are located upstream of the sensors on the return leg of the leaching solution circulation loop. This is where acetic acid or other reagents are added to the leaching solution by peristaltic pumps. Figure 14 shows the set of three pumps, one of which is a backup.

Figure 13. Chemical injection location and chemistry sensors.

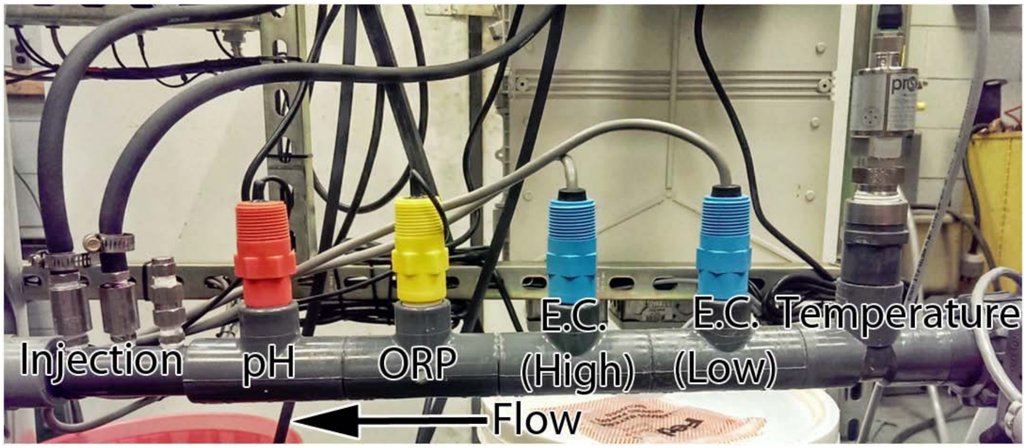


Figure 14. Peristaltic metering pumps.

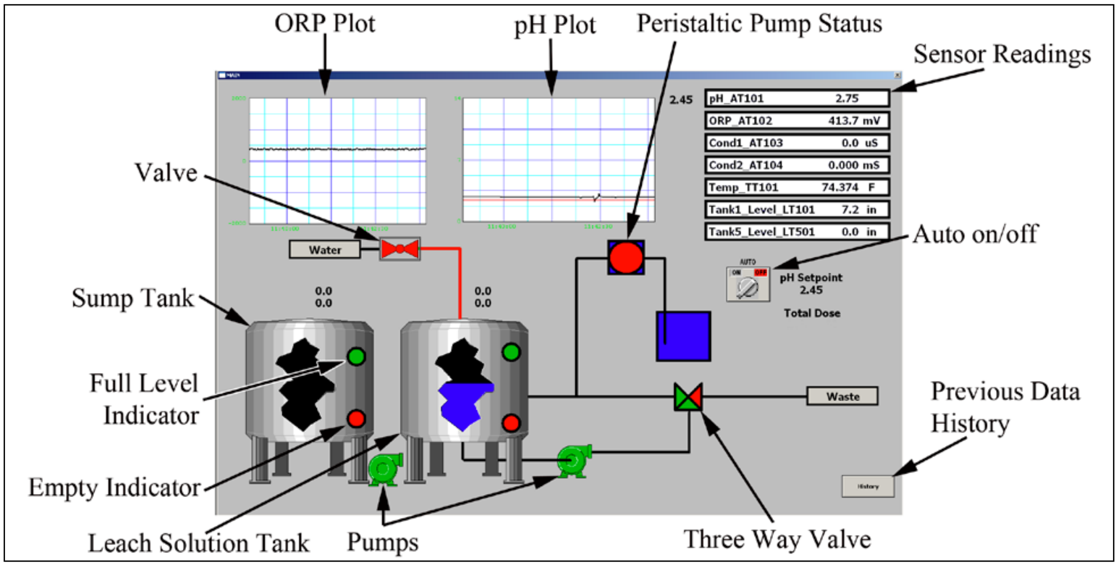
The leaching process uses a combination of acetic acid, hydroxylamine hydrochloride, and water. Water is pumped into the leaching solution tank from a water source and is regulated by a solenoid valve. Figure 15 shows water being added through the pipe on the left side of the tank. A food-grade color concentrate is added to the water to evaluate effectiveness of tank mixing. The photo in Figure 16 was taken approximately 2 seconds after the food color was added and shows how uniformly tank contents are mixed.

Figure 15. Water being added to the batch mixing tank.

Figure 16. Mixing of food-grade color concentrate in batch mixing tank.

The SCADA monitors the pH of the leaching solution in the leaching solution tank as acetic acid is being added. A photograph with descriptors of the SCADA GUI is provided in Figure 17. A *green* status indicates that pumps are on and valves are open. A *red* status indicates that valves are closed and pumps are off. *Green* on the tank level indicators indicates that the fluid level is below the sensor on the tank. The color *blue* inside tank icons provides an estimate of the level of the fluid relative to the two fluid level sensors. Tank fluid levels intermediate to the high and low sensors are determined using differential pressure sensors previously described. The three-way valve has three states: recirculating, transfer, or off. The three-way valve is set to recirculating when the left and bottom triangles are green. It is set to transfer when the right and bottom triangles are green. The three-way valve is off when all three are red.

Figure 17. Screenshot of supervisory control and data acquisition GUI with descriptors.



A manual ball valve can be seen in Figure 18. This valve is used to control the rate at which leaching solution flows from the leaching solution tank to the leach heap. There is a second three-way actuated valve not shown in the SCADA software that is detailed in Figure 18. While the valve is not displayed in the SCADA GUI, it is monitored by SCADA software. The valve is controlled by system software and directs fluids to leaching or wasting depending on the leaching chemistry, particularly electrical conductivity. System software transfers the fluid to the waste tank when solution conductivity reaches the setpoint signaling that it has become exhausted. A flow diagram of the two different transferring states (leaching or waste) can be seen in Figure 19.

Figure 18. Actuator valve for leaching or transferring to waste storage.

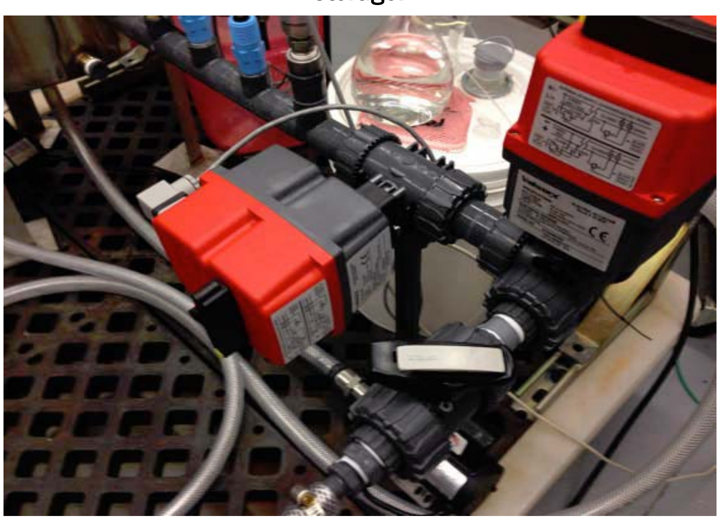


Figure 19. Transfer leach or waste flow depiction.

Soil to be remediated is placed into the heap vessel. The heap vessel is located above the sump tank in order to take advantage of gravitational transfer. A photograph of the leach heap tank can be seen in Figure 20. The leach heap vessel has three primary components: spray bar, level switch, and false bottom. A photograph of the interior of the vessel, including the spray bar, level switch, and false bottom, can be seen in Figure 21. The spray bar has the spray holes pointed up for easy viewing as opposed to being directed downward as they would be during use. A design diagram of the leach heap vessel can be seen in Figure 22 showing the geometry of the leach heap.

Figure 20. Photograph of leach heap tank.

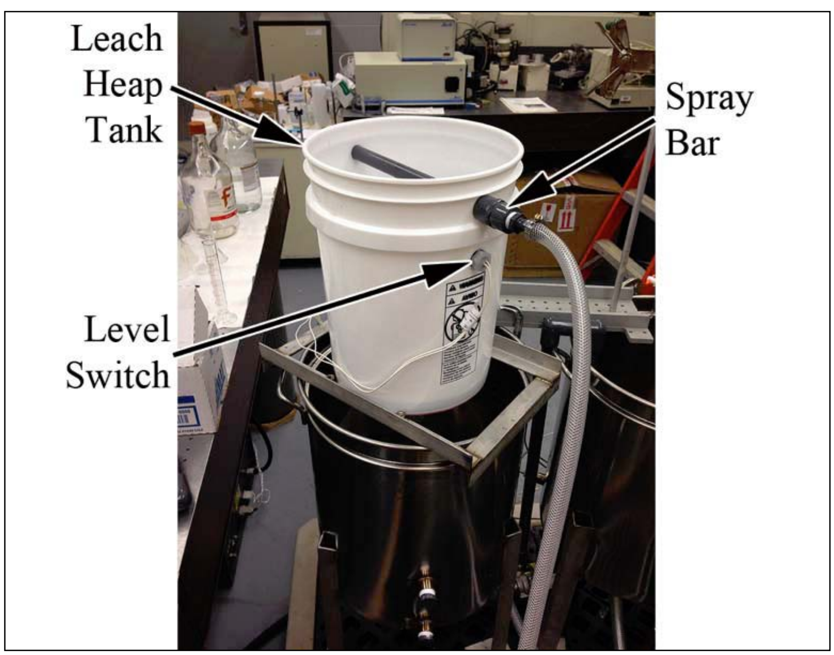
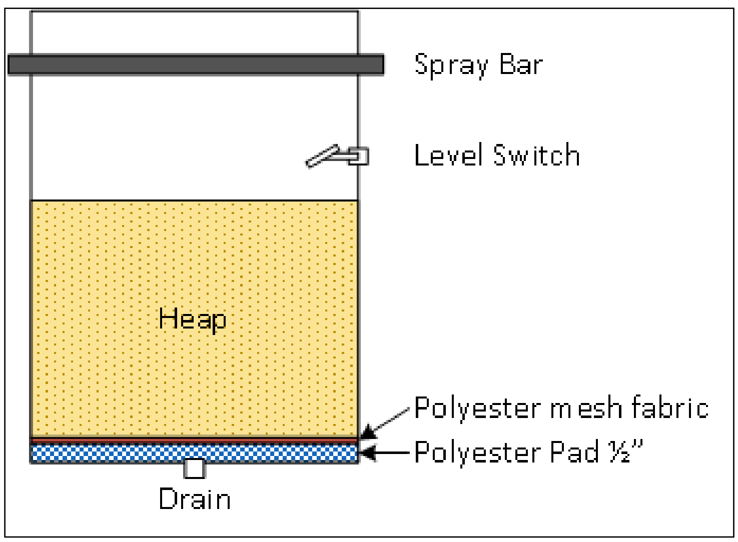


Figure 21. Interior of the leach heap tank. Spray bar was turned to show detail.

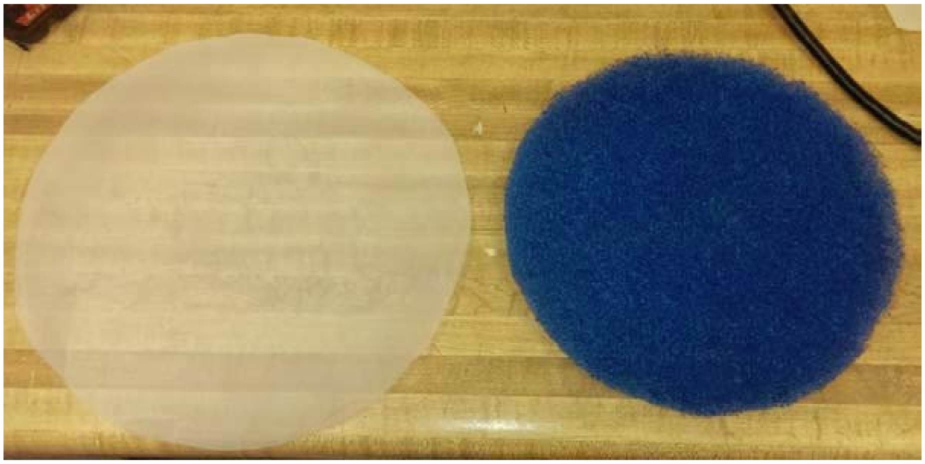
Figure 22. Design drawing of leach heap tank.



The false-bottom screen is constructed of a polyester mesh fabric and half-inch-thick polyester padding. This simulates a thick pad of filter fabric in a full-scale application. Individual components of the false-bottom can be seen in Figure 23.

Soil designated for remediation is placed on top of the false bottom in the leach heap vessel and is covered with leaching solution. A weep hose can be used in place of the spray bar to distribute the leaching solution beneath the soil surface. Even distribution of leachate promotes and aids in maintaining vertical seepage of leachate through the entire depth of the heap. This also reduces the potential of forming preferential flow paths and creating dead spots within the heap where leaching is not occurring.

Figure 23. Components of the leach heap tank false bottom.



The leachate seeps through the false bottom and is collected in the sump tank under the heap. The uranium-containing leachate passes through a filtration unit to remove suspended solids and then through the apatite column to remove the uranium. Differential pressure across the filtration units will be monitored to determine when cleaning is needed. Additionally, leachate will be monitored using lanthanum bromide gamma detectors before it passes through the apatite column and again as it exits. This will provide data for estimating the activity of uranyl materials collected by the column and also indicate when the apatite has become exhausted.

4 System Performance

4.1 Soil leaching pH stabilization

The soil leaching system's ability to monitor and adjust the soil leaching solution was evaluated. Testing began with calibration of all sensors. The pH, ORP, and conductivity sensors were calibrated using calibration solutions from Hanna Instruments, Inc. (http:hannainst.com/usa). The pH sensor was calibrated using solutions buffer standards of pH 4.01, pH 7.01, and pH 10.01. The ORP sensor was calibrated using a standard 470 mV solution. Conductivity sensors were calibrated using 84 µS/cm and 111800 µS/cm standards. Each sensor was removed from the system and placed into the calibration solutions. The sensor transmitters are equipped with calibration potentiometers that were adjusted for each calibration.

A standard leaching solution of 5% acetic acid was produced with a pH of 2.4. Solutions of sodium hydroxide were added to leaching solution to determine the ability of the SCADA to monitor pH and then to return the solution to the pH setpoint.

A screenshot of the SCADA software can be seen in Figure 24. The SCADA software has a variety of functions: monitoring pH, ORP, conductivity, solution volumes, turning on pumps, valves, recording the volume of acetic acid added to solutions. Two pump icons can be seen at the bottom of the screen shot, one green and one red. Pumps are activated or deactivated by clicking on the pump icon. Valves are turned on or off or changed in direction also by clicking on the icon on the screen. Water is being added to the batch-mixing tank in Figure 24, as an example.

Twenty liters of 5% acetic acid leaching solution were made for this evaluation. Thirty-eight liters of water were added to leaching system. Two liters of analytical grade glacial acetic acid were added to the solution. The pH of the solution was tested using a Scientific Products Model 8025pH meter to verify accuracy of the system's pH measurement.

Figure 24. Screenshot of SCADA software control.

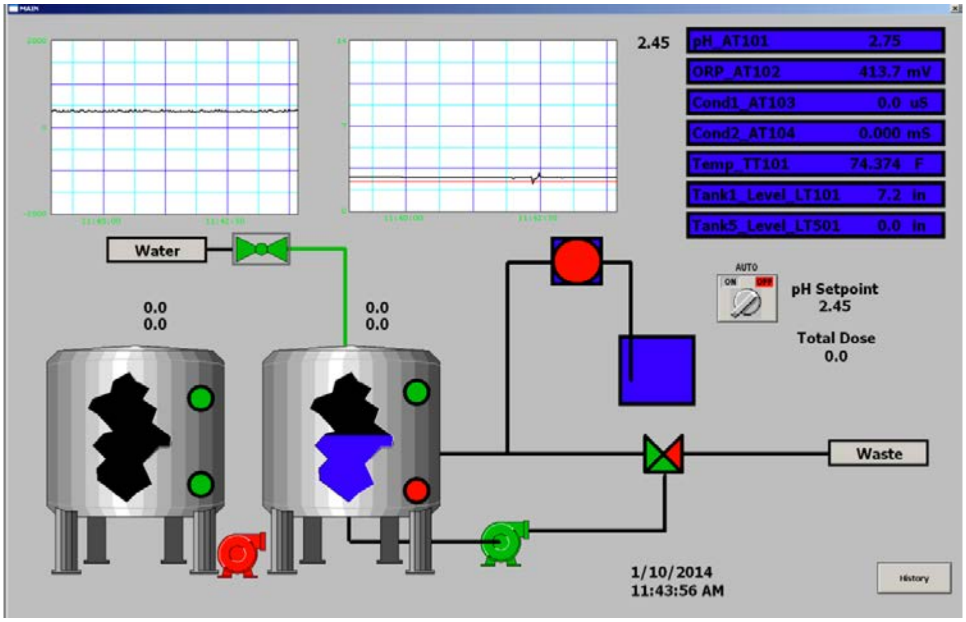


Figure 25 is a screenshot of the configuration during the SCADA-controlled tank mixing. The batch-mixing tank pump is turned on, the two-way pump is set to return the solution to the batch tank, and the water valve is turned off. Figure 26 is a photograph of the mixing process.

Figure 25. SCADA settings for mixing the solution.

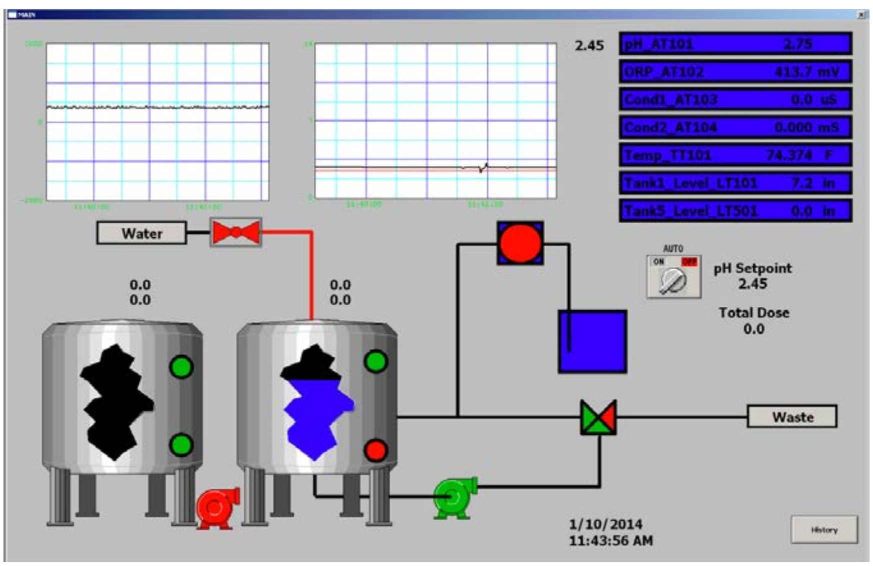
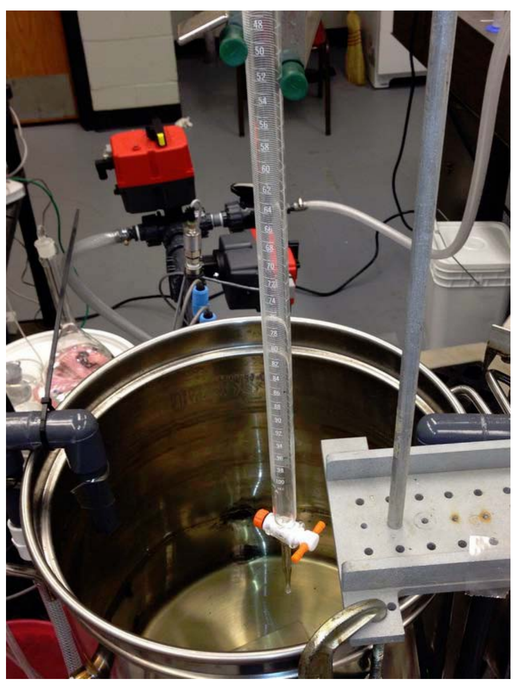


Figure 26. Photograph of 5% acetic acid solution in the leaching system.

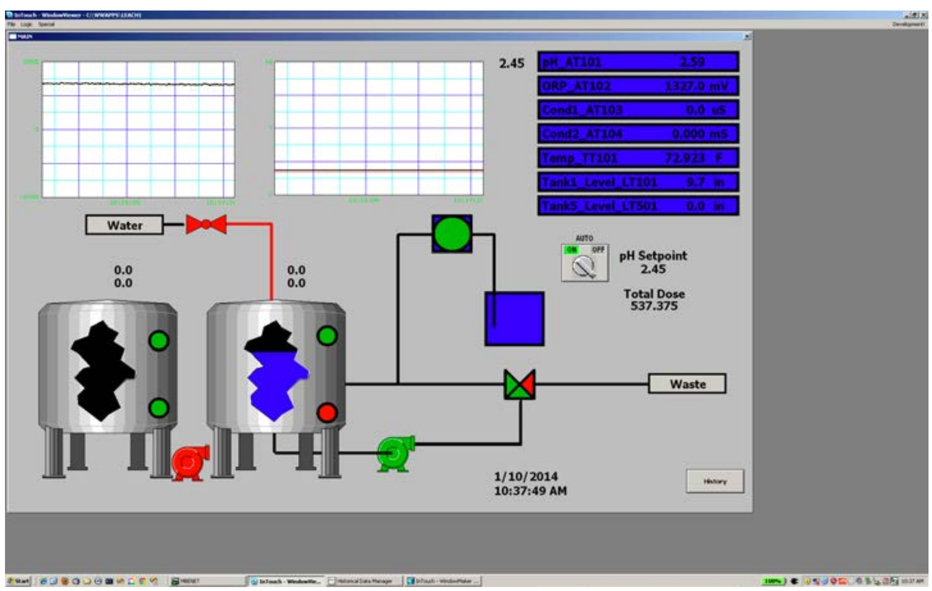
A solution of 1.5 M sodium hydroxide was used to increase the pH of the leaching solution. A 100-milliliter (ml) graduated burette was used to introduce the sodium hydroxide solution to the leaching solution while mixing was occurring. The graduated burette was mounted above the batch tank (Figure 27).

Figure 27. Graduated burette used for the introduction of sodium hydroxide.



Five volumes of 1.5 M sodium hydroxide were added to the leaching solution: 6 ml, 14 ml, 30 ml, 50 ml, and 50 ml for a total of 150 ml. Each addition increased the pH of the leaching solution. The 150 ml of sodium hydroxide brought the final pH to approximately 2.63. The SCADA software was programmed to adjust the chemistry of the leaching solution to maintain a pH of 2.45. A screen shot of the SCADA system setting can be seen in Figure 28. Clicking on the auto-on-off switch next to the pH setpoint notification turns on the software's auto-pH adjustment setting. The SCADA software monitors the pH of the solution and makes adjustments by using a peristaltic pump to introduce more acetic acid. The green-colored circle to the left of auto-on-off switch indicates activation of the peristaltic pump.

Figure 28. Screenshot of SCADA software set to adjust solution pH.



The SCADA software recognized the increased pH and began pumping a 10% acetic acid solution into the leaching solution. The 10% acetic acid solution was switched to glacial acetic acid shortly after it was recognized that the 10% solution would take too long to re-establish the setpoint pH. Figure 29 shows a plot of the pH as a function of time with annotations of the regions of interest. Figure 30 shows a plot of the pH as a function of time along with the total volume of sodium hydroxide as a function of time.

Figure 29. Plot of leaching solution pH as a function of time as the SCADA system automatically adjusts pH.

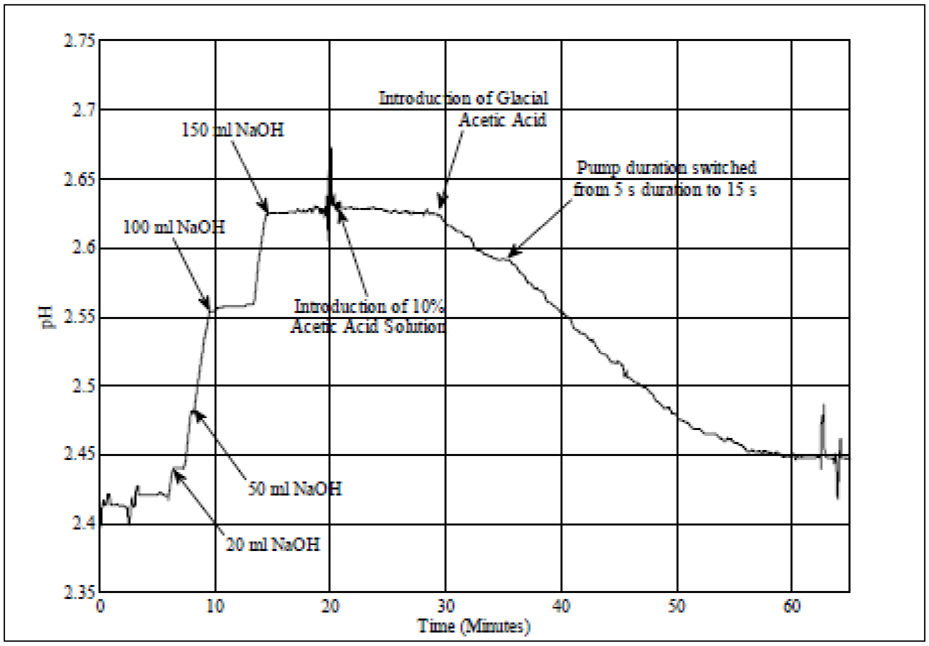


Figure 30. Total volume of acetic acid and pH as a function of time.

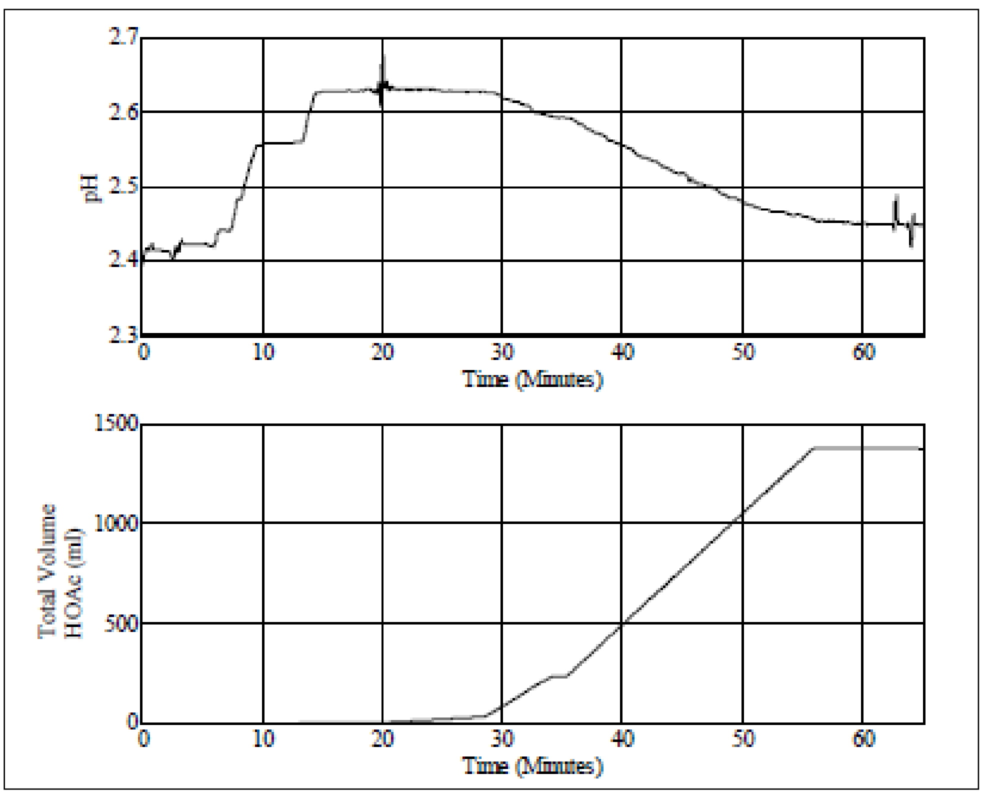
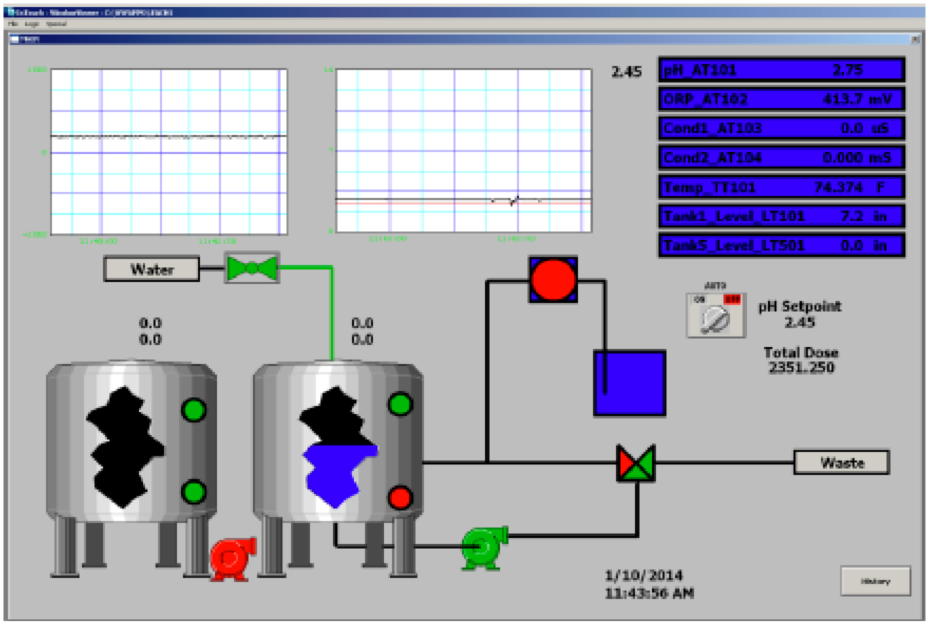


Figure 31 shows an example screenshot of the solution being pumped to waste storage. Water was also added during the draining process. The batch tank was filled with clean water after draining. Clean water was circulated through the system to clear acetic acid that was not removed during the draining process. The circulated water also ensures that sensors are rinsed. This water was drained, and the rinsing cycle repeated.

Figure 31. Screenshot of SCADA software transferring solution to drain.



Between the two-way valve and the drain is another manual two-way valve that is not shown in Figure 31. This valve allows users to flood the top of the heap with leaching solution. Leachate is caught in the sump tank. The volume of the sump tank (left tank) is monitored in the SCADA software. The postleaching solution can be transferred back to the batch-mixing tank by clicking on the red pump icon located between the two tanks in the SCADA software. Figure 32 shows a screenshot of the settings used to transfer postleaching solution from sump tank to batch-mixing tank.

4.2 Radiation detection

A radiation detection system has been designed and developed to estimate the amount of DU within a solution. The system consists of a cerium-doped lanthanum bromide scintillation detector located in a cavity that is surrounded by tubing in the shape of a helix. The scintillation detector is used to measure the gamma rays emitted from fluid located in the tubing. The tubing is used to carry leaching solutions containing uranium from the

bottom of the leach heap to the detector and back to the tank using a peristaltic pump. Figure 33 shows a photograph of the tubing and detector cavity. Figure 34 shows a photograph of the detector system.

Figure 32. SCADA software settings for transferring postleaching solution to the batch mixing tank.

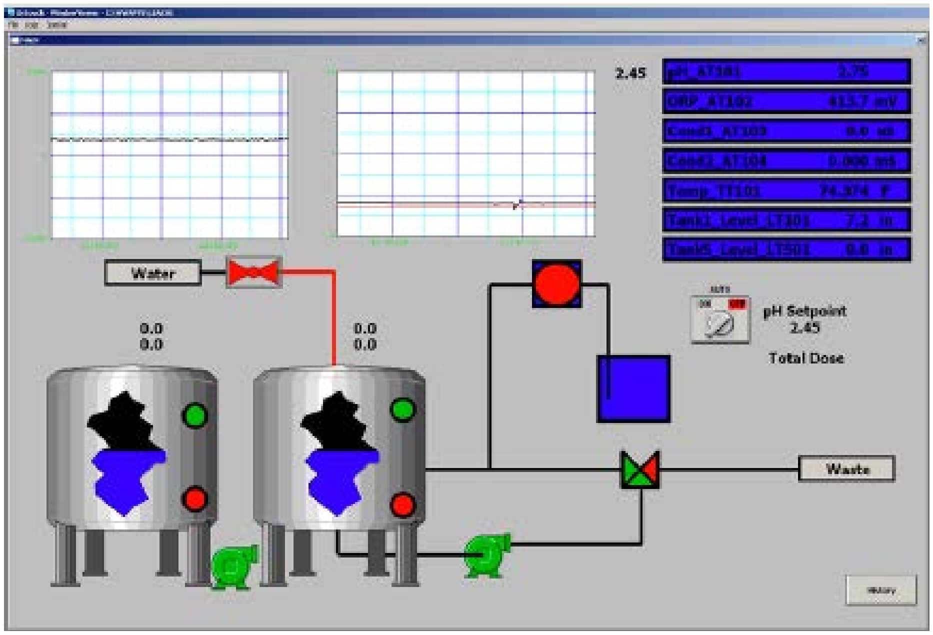


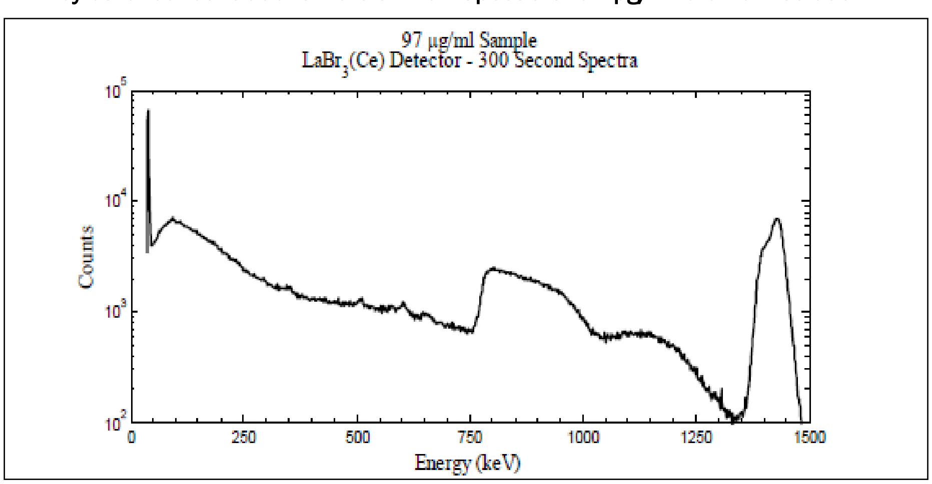
Figure 33. Interior of the detector system showing tubing and the detector cavity.

Figure 34. Photograph of the heap leaching detector system.

A stock solution of uranyl acetate in water was produced for evaluating the performance of the radiation detection system. Uranyl acetate is composed of 65.01% depleted uranium. A solution of 200 ml was made with 4.9187g of uranyl acetate. The stock solution had 16.2 mg/ml of uranium, 0.0062 µCi/ml of activity, and the entire solution had an activity of 1.26 µCi.

The stock solution was used to produce three solutions with different activities. The following uranium concentration solutions were made: 97 µg/ml, 130 µg/ml, and 510 µg/ml. Each solution was employed for a detection sensitivity study using a peristaltic pump to fill the tubing. Gamma ray spectra were collected for each solution 10 times. Figure 35 is an example of spectra collected for each solution.

Figure 35. Example of the spectra of 97 ug/L uranium solution. The spectra provided by other concentrations were similar. Spectra of 97 µg/ml uranium solution.



These spectra are only marginally different from background spectra. However, they do provide useful counting statistics to demonstrate the increased rate at which gamma rays are detected by the lanthanum bromide detector. All three samples displayed statistically significant increased counts above background (Table 2).

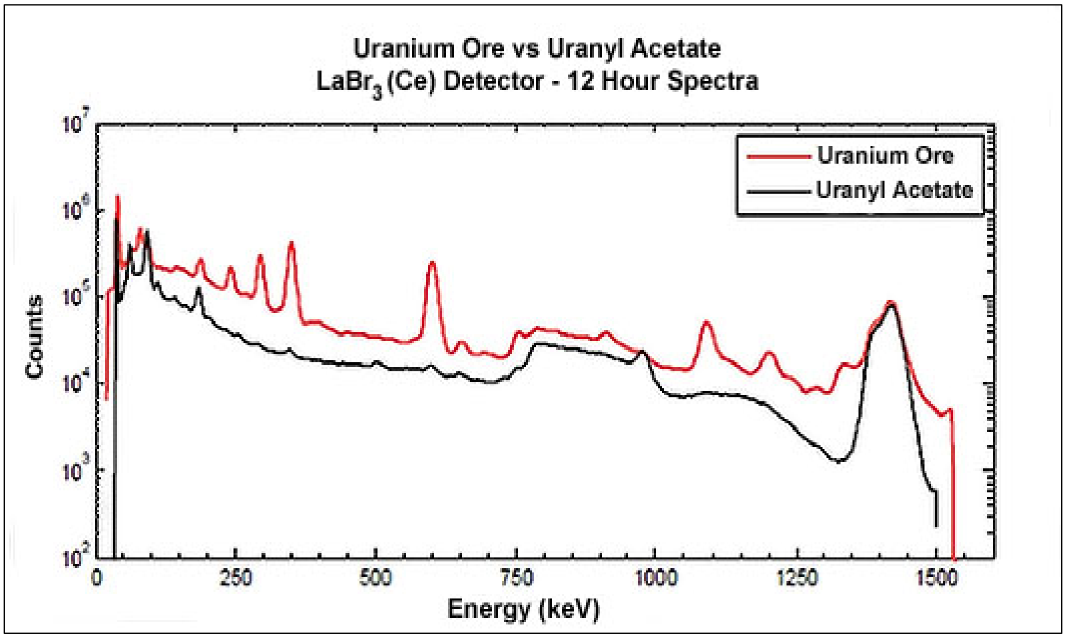
Table 2. Counting statistics from 1-hour gamma ray spectra.

Sample	Counts (counts /hr, cph)	Difference (cph)
Background	1,850,311	—
97 µg/ml	1,894,848	34,537
130 µg/ml	1,894,091	43,780
510 µg/ml	2,001,684	151,373

The concentration of uranium in solution can be estimated based on the counting statistics above. However, background radiation from soil is different from location to location. The specific activity of uranium and DU are also dependent on the ratios of 238U, 235U, and 234U in the sample.

Natural uranium (NU) is composed of 235U, 238U, and a trace of 234U. In terms of the amount of radioactivity, approximately 2.2% comes from 235U. The majority of the radiation is split between the other isotopes: 48.6% from 238U and 49.2% from 234U. The 238U in NU also contains daughters that are radioactive. This increases the activity of samples over long periods of time. A few of the 238U daughters are gamma emitters. Samples of DU are virtually devoid of these gamma-emitting daughters due to long half lives of decay-chain members. This difference in gamma emitters in samples can be used to discriminate DU from NU. Spectra in Figure 36 show the difference in the gamma spectrum from DU (black) and NU (red).

Figure 36. Gamma ray spectra comparing DU and NU.



5 Conclusions

In summary, the goal of this project was the development of a small-scale, yet highly scalable SCADA system. This system can be used for evaluating/improving performance of the ERDC-EL soil washing process that removes DU sorbed to soil fines. This system can facilitate scale up for field use. The prototype soil leaching SCADA system has been demonstrated to meet the fundamental design and performance requirements discussed in Chapters 2 and 3 of this report, concerning automatic control of the following functions:

- leach solution pH, ORP, and conductivity
- radiation detection
- flow
- leachate transfer
- heap pile and piping rinse
- waste transfer.

The SCADA was demonstrated to control pumps and valves, collect chemical parameter data and generate trend lines, monitor and estimate tank fluid volumes, and exercise chemical control of leaching solutions. Budget limitations prevented development of a more complicated leaching system to evaluate performance with leaching of soil samples containing depleted uranium.

The control system has employed hardware and software packages capable of scaling to much larger and more complicated applications. Additional programmable logic controllers can be employed by the SCADA, and WonderWare has been used for networking much larger and more complicated control systems.

The SCADA and system hardware can be easily adapted to conducting research activities to optimize the ERDC-EL DU soil washing process. Current hardware can be duplicated to service an array of 1-cubic-foot leaching vessels for conducting parallel testing with the same or different leaching solutions. The system can also be expanded to operate a large-scale leaching system utilizing thousand-gallon tanks and heaps of hundreds of cubic yards of contaminated soil.

Sensors used to control chemistry of the leaching solutions need to be industrial quality. However, the most difficult measurements will be those determining uranium activity levels in leachate before and after the Phase 2 adsorption column. Unfortunately, detection sensitivity and selectivity will be the most expensive components of the system. Lanthanum bromide detectors provide good sensitivity and energy resolution, and a pair of these units plus multichannel analyzers will cost approximately $100,000. Less expensive detectors can be used, but spectral ratios generated from these units are less specific. Spectral data provided in Chapter 4 emphasize the need for high resolution capability.

References

Larson, S. L., V. F. Medina, J. H. Ballard, C. S. Griggs, M. Wynter, D. Mackie, B. King, and C. Nestler. 2012. *Large-scale physical separation of depleted uranium from soil.* ERDC/EL TR-12-25. Vicksburg, MS: U.S. Army Engineer Research and Development Center.

Larson, S., J. Ballard, V. Medina, M. Thompson, G. O'Connor, C. Griggs, and C. Nestler. 2009. *Separation of depleted uranium from soil.* ERDC/EL TR-09-1. Vicksburg, MS: U.S. Army Engineer Research and Development Center.

Monts, D. L., G. Wang, Y. Su, P-R. Jang, and C. A. Waggoner. 2009. *Development of fluoresence spectral imaging for location of uranium deposited on surfaces.* Paper 9163, Waste Management Conference, March 1–5, 2009, Phoenix, AZ.

Walters, J., J. Mackey, K. Maki, R. Northcutt, J. V. Farr, W. A. Martin, S. L. Larson, J. H. Ballard, and C. Nestler. 2014. *Cost analysis of remediation systems for depleted uranium*. ERDC/EL TR-14-5. Vicksburg, MS: U.S. Army Engineer Research and Development Center,

Wang, G., Y. Su, and D. L. Monts. 2008. Parametric investigation of laser-induced fluoresence of solid-state uranyl compounds. *The Journal of Physical Chemistry A* 112: 10502–10508.

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14. ABSTRACT
The U.S. Army has responsibility for maintaining or managing a large number of facilities that are or have been used for training troops and developing/testing equipment and munitions, including ranges that may have been contaminated with uranium. Licenses issued by the Nuclear Regulatory Commission (NRC) for use of radiological materials such as depleted uranium (DU) specify the isotopes that can be used, along with possession limits for the site. U.S. Army Engineer Research and Development Center (ERDC) researchers have developed a soil washing system to leach DU oxides from soil. The Institute for Clean Energy Technology (ICET) at Mississippi State University (MSU) has developed an effective survey system to accurately locate areas of DU contamination for removal and disposal. The ICET also has a history of developing control systems for sophisticated test beds. ICET has combined its experience in development of control systems with DU detection methods to develop a process control system for the ERDC soil leaching system for extracting DU from contaminated range soil. The ICET system control and data acquisition (SCADA) system has been demonstrated to control pumps and valves, maintain leaching solution chemistry to user-defined set points, and detect environmental levels of DU oxides in leachate. The SCADA system will assist the ERDC Environmental Laboratory (EL) in transitioning development of the soil washing system from pilot to a full-scale system.

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